

5(3)

AUTHORS:

Kochetkov, N. K., Nifant'ev, E. Ye., Molodtsov, L. V.

TITLE:

Bromination of β -Ketoacetals (Bromirovaniye β -ketoatssetal'ev)

PERIODICAL:

Zhurnal obshchey khimii, 1962, Vol. 32, No. 7, p. 2330-2337 (USSR)

ABSTRACT:

Hitherto the easily accessible β -ketoacetals $ROOCH_2CH(OR')_2$ were used either as potential β -dicarbonyl compounds (Refs 1, 4), seeing they resemble the β -chloroalkylketones in their reactions or as ketones having another reactive substituent. A third possibility, i.e. that of introducing substituents into the central methylene group of the ketoacetal, has so far been disregarded, even though the resulting compounds could be utilized for synthesis in various directions (Ref 8). As first reaction of this kind the hitherto unknown bromination of the ketoacetals was undertaken. Thus, two methods were elaborated, one for the synthesis of α -bromo- β -ketoaldehydes by bromination of β -ketoaldehydes in aqueous solution in the presence of barium carbonate, and another for the preparation of ethylene acetals of α -bromo- β -ketoaldehydes by bromination of the ethylene acetals of β -ketoaldehydes in ether in the presence of barium carbonate. By condensation of α -bromo- β -ketoaldehydes

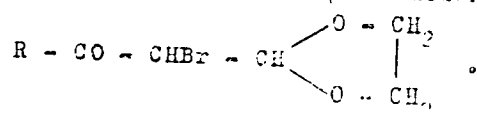
Card 1/2

Bromination of β -Ketoacetals

SOV/73-20-7-49/

with urea, the 2-amino-5-acyloxazoles were obtained; on condensing with thiourea and thioamides of acids 2-substituted 5-acylthiazoles were formed. Thus, it was demonstrated, that the α -bromo- β -ketoaldehydes react similarly to α -bromoaldehydes in the reactions under investigation. On treating ethylene acetals of α -bromo- β -ketoaldehydes with β -naphthol in the presence of iron chloride and hydrochloric acid 2-alkyl-3-bromo-naphtho-(1,2;5',6')-pyryl salts were formed. Table 1 shows the synthesis of the α -bromo- β -ketoaldehydes:

$R-CO-CHBr-\overset{\overset{O}{\parallel}}{C}-H$ and table 2 the synthesis of the ethylene acetals of α -bromo- β -ketoaldehydes:



There are 2 tables and 15 references, 6 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)
SUBMITTED: June 5, 1958
Card 2/2

5(3)

SOV/79-29-8-24/81

AUTHORS:

Kochetkov, N. K., Nifant'yev, E. Ye., Sokolov, S. D.

TITLE:

Synthesis of Aromatic β -Ketoacetals

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 8, pp 2570-2575
(USSR)

ABSTRACT:

The β -ketoacetals which are mainly obtained from the corresponding β -chloro-vinyl ketones (Ref 1) are recently of manifold use in organic synthesis, whereas compounds of this class with aromatic radicals have nearly escaped notice (Refs 2-5). Quite recently, the authors synthesized the ethylene acetal of the benzoyl-acetaldehyde (Ref 6). Presently, they carried out the synthesis of various aryl- β -ketoacetals using, as initial products, the acyl- β -chloro-vinyl ketones which are now well accessible (Ref 7). Three new representatives of this class, the o-tolyl- and m-tolyl- β -chloro-vinyl ketone, and the n-bromo-phenyl- β -chloro-vinyl ketone, were synthesized. On the first attempts to obtain the acetal of benzoyl-acetaldehyde according to reference 1, the phenyl- β -methoxy-vinyl ketone (II) was formed instead of the expected compound. This was due to the catalytic action of alkali traces left over in the re-

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SOV/79-29-8-24/81

Synthesis of Aromatic β -Ketoacetals

action. Therefore, the reaction conditions were altered in such a way that in the distillation any traces of alkali were excluded by treating the reaction mixture with water, and extracting with ether. Thus the acetal of the benzoyl-acetaldehyde (III) resulted in a 60% yield. More convenient are the ethylene acetals of the β -ketoaldehydes which were recently synthesized by the authors (Ref 6). From among the representatives of the aromatic series, only the ethylene acetal of the benzoyl-acetaldehyde (IV) is described in publications. The fact that the cyclic ethylene acetals, which can easily be obtained by reaction of β -chloro-vinyl ketones with ethylene glycol, are formed by treating both the dimethyl acetals and the alkoxy-vinyl ketones with ethylene glycol in the presence of alkali, indicates their considerably higher stability. All transformations described, which are connected with the investigation of the stability of the acetals of the benzoyl-acetaldehyde, are illustrated in scheme 1. KOH and K_2CO_3

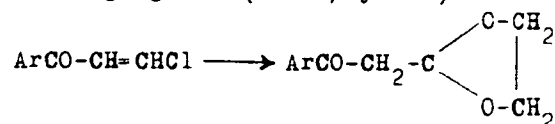
(Ref 6) proved to be the agents most useful for condensing the ethylene acetals of the β -ketoaldehydes of the aliphatic series. The operational method devised for the synthesis of the first member of the series was further applied to the syn-

Card 2/3

Synthesis of Aromatic β -Ketoacetals

SOV, 79-29-8-24, 8.

thesis of the ethylene acetals of the β -ketoaldehydes substituted in the aromatic nucleus, using KOH and K_2CO_3 as condensing agents (60-80% yield):



where Ar = n,m,o- $\text{CH}_3\text{C}_6\text{H}_5$; n,o- ClC_6H_4 ; n- BrC_6H_4 ; n- $\text{CH}_3\text{o-C}_6\text{H}_4$.

The resultant crystalline acetals are stable, in general well soluble, and do not color with ferric chloride. There are 1 table and 14 references, 6 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 3, 1958

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5(3)

AUTHORS:

Kochetkov, N. K. Nifant'ev, E. Ye.
Kulakov, V. N.

SOV/20-125-2-24/64

TITLE:

Synthesis of β Ketomercaptals (Sintez β -ketomerkaptaley)

PERIODICAL:

Doklady Akademii nauk SSSR 1959, Vol 125, Nr 2, pp 327-329
(USSR)

ABSTRACT:

The preparative use of β ketoacetals (Refs 1, 2) which can be obtained readily and with good yields from the interaction with alcohols and glycol of β chlorovinylketones in an alkaline medium is rendered difficult by their very marked tendency towards hydrolysis in acid media. For this reason, the synthesis of the sulfurous analogues of the β ketoacetals, i. e. of the substances mentioned in the title, was attempted. It was known that the mercaptal group is sufficiently stable in the acid medium (Ref 3). In view of the existing difficulties in the synthesis of oxy methylene ketones (initial substances), the authors have developed a convenient general synthesis method for β ketomercaptals by means of ketovinylation of mercaptans (yields 50-90%). This reaction occurs quite readily in an aqueous solution in the presence of potash. As in the cases of the alcohols and of glycol (Refs 1, 2), and unlike

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Synthesis of β -Ketomercaptals

SOV/20 125-2-24/64

the processes taking place in the cases of the phenols (Ref 5) and thiophenols (Ref 6) the reaction does not stop after the substitution of the chlorine atom in the chlorovinylketone but is completed by the attachment of the second mercaptan molecule to the double bond. This is how mercaptal is formed. This reaction has a general character. On the one hand, this reaction is entered into by β -chlorovinylketones both with aliphatic and with aromatic radicals on the other hand it is entered into by both monatomic and diatomic mercaptane. For this purpose, the sulfurous analogue of ethylene glycol, 1,2-ethane-dithiol (Ref 7) appears most appropriate. The aliphatic β -ketomercaptals thus produced are stable oily liquids, their analogues with aromatic radicals are solid well crystallizable substances. The ketomercaptals enter into such reactions as are typical of the β -ketoaldehydes, which sufficiently proves their structure. They oxidize readily into the corresponding disulfones (with perhydrol in HCl according to reference 8). These disulfones have a marked tendency towards hydrolytic decomposition in an alkaline medium. These reactions can be of interest for the production

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Synthesis of β -Ketomercaptals

SOV/20-125-2-24/64

of various oxy-methyl-ketones. The experimental part contains the usual data. There are 2 tables and 13 references, 8 of which are Soviet.

PRESENTED: December 1, 1958, by A. N. Nesmeyanov, Academician

SUBMITTED: November 29, 1958

Card 3/3

Phosphorus-containing Polymers. I.
Synthesis and Polymerization of Ethylene
Alkyl Phosphates

81585
S/190/60/000/02/00
B020/B066

polymerization under the same conditions (with a degree of polymerization of 10 - 14). The resultant polymers had about the same molecular weight and the same properties as found in experiments without or with catalysts (sodium). High-molecular phosphates are neutral substances with polyester structure. The high-molecular phosphates described in this paper may be used as plasticizers.¹⁹ The experimental part describes the preparation of ethylene hexyl phosphite, ethylene phenyl phosphite, and ethylene alkyl phosphates (Table 1), the conditions of polymerization of ethylene alkyl phosphates and the properties of the polymers (Table 2) as well as the reaction of polyethylene propyl phosphate with phosphorus pentachloride. It is shown that ethylene alkyl phosphates polymerize on prolonged standing or heating. It was also found that the yield of ethylene alkyl phosphates obtained by the above-described process is 55 - 83 % , and that on polymerization the latter form polyesters (molecular weight of 2,000 - 3,000) by cleavage of the cyclic phosphates. There are 2 tables and 5 references: 3 Soviet, 1 US, and 1 British.

SUBMITTED: December 14, 1959

Card 2/2

83817

S/190/60/002/005/007/C
B004/B067

2109
2209

15.8114

AUTHORS: Petrov, K. A., Nifant'ev, E. Ye., Sopikova, I. I.
TITLE: Phosphorous Polymers. II. Use of the Arbuzov Rearrangement
for Synthesizing Polyphosphonates
PERIODICAL: Vysokomolekulyarnyye soyedineniya, 1960, Vol. 2, No. 1,
pp. 685-688

TEXT: The authors used the Arbuzov reaction for synthesizing polymer phosphinic esters. Polymerization occurs on heating 1 mole of cyclic phosphinites with 0.001 - 0.1 mole of methyl iodide in a sealed tube. Polyphosphinites are formed with a molecular weight of 270 - 3200. The hitherto unknown cyclic phosphinites were obtained by reacting dichlorophosphines with 1,3-diols in the presence of tertiary amines: phenyl-(1,3-butylene) phosphinite and phenyl-(1,3-propylene) phosphinite. The reaction with CH_3I is a recurring alkylation reaction with

Card 1/2

83817

Phosphorous Polymers. II. Use of the
Arbuzov Rearrangement for Synthesizing
Polyphosphonates

S/190/60/002/005/007/011
B004/B067

$$\begin{array}{c} \text{R} \\ \diagup \\ \text{P}=\text{O} \\ \diagdown \\ \text{CH}_3 \end{array} - \text{O} \left[-(\text{CH}_2)_n - \begin{array}{c} \text{O} \\ \diagup \\ \text{P} \\ \diagdown \\ \text{R} \end{array} - \text{O} \right]_m - (\text{CH}_2)_n - \text{I}$$
 as final product. The structure of the

central member could be proved by cleaving the phenyl-(1,3-butylene) phosphinite polymer by means of phosphorus pentachloride. Phenyl (1-methyl-3-chloropropyl)phosphinyl chloride was obtained. The results of polymerization of phenyl-(1,3-butylene) phosphinite with various additions of methyl iodide are given in a table. There are 1 table and 2 references: 1 Soviet and 1 US.

SUBMITTED: January 15, 1960

Card 2/2

5.3600

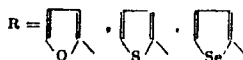
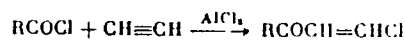
11393
307/19-10-1-1-1/12

AUTHORS: Kochetkov, N. K., Nifant'yev, E. Ye., Nifant'yeva, L. V.

TITLE: β -Chlorovinyl Ketones of the Heterocyclic Series

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, No 1, pp 241-245 (USSR)

ABSTRACT: Synthesis of some β -chlorovinyl ketones, containing a five-membered heterocyclic radical, by the condensation of the corresponding acid chlorides with acetylene, was studied. It was found that acid chlorides of furan-2-carboxylic, thiophene-2-carboxylic, and selenophene-2-carboxylic acids easily condense with acetylene to form corresponding β -chlorovinyl ketones:

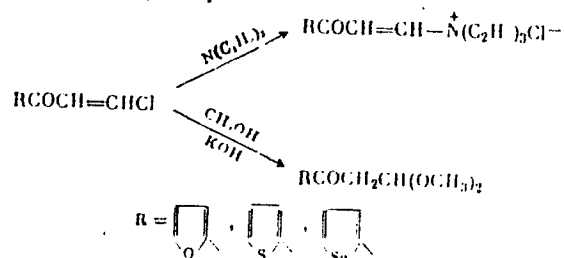


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β -Chlorovinyl Ketones of the Heterocyclic Series

77333
SOV/10-50-143-713

The reaction takes place at 30-40°. The heterocyclic β -chlorovinyl ketones, like other vinyl ketones, react with alcohol in the presence of alkalis to form β -keto-acetals:

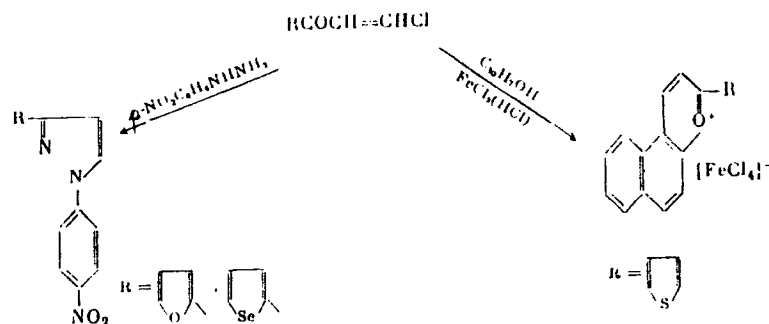


They also readily condense with p-NO₂C₆H₄NHNH₂ to form corresponding pyrazole derivatives. Thienyl-(α)- β -chlorovinyl ketone condenses with β -naphthol in the presence of ferric chloride and HCl.

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β -Chlorovinyl Ketones of the Heterocyclic Series

77-26
SC: 77-26-1-1-1-1-1-1



Preparation of the following compounds is given:
 Furyl-(2)- β -chlorovinyl ketone (41%, based on acid chloride), bp 102-105° (10 mm). Thienyl-(2)- β -chlorovinyl ketone (65%), bp 154-156.5° (23 mm). Selenyl-(2)- β -chlorovinyl ketone (45%), bp 132-135° (7 mm).

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β -Chlorovinyl Ketones of the Heterocyclic Series

77333
S07/79-30-1-5-78

Dimethyl acetal of furoyl-(2)-acetaldehyde (6%), bp 122-123° (10 mm), n_D^{20} 1.4998, d_4^{20} 1.1800. Dimethyl acetyl of thienoyl-(2)-acetaldehyde (53%), bp 1-7-148° (8 mm), n_D^{20} 1.5146, d_4^{20} 1.1910. 3-Furyl-(2')-1-(p-nitrophenyl)-pyrazole (62%), mp 70.5-72°. 3-Selenyl-(2')-1-(p-nitrophenyl)-pyrazole (63%), mp 100-101°. 2-Thienyl-(2')-naphtho-(1,2:5,6)-pyrylium ferrichloride (66%), mp 176-177°. There are 11 Soviet references.

ASSOCIATION: Moscow State University (Moskovskiy gosudarstvennyy universitet)

SUBMITTED: September 30, 1958

Card 4/4

S/079/60/030/006/032/033/XX
BOO/BO55

AUTHORS: Kochetkov N K and Nifant'yev. E. Ye

TITLE: Oxidation¹ of β -Ketoacetals¹ by Means of Lead Tetraacetate

PERIODICAL: Zhurnal obshchey khimii, 1960 Vol 30 No. 6,
pp. 1866 - 1872

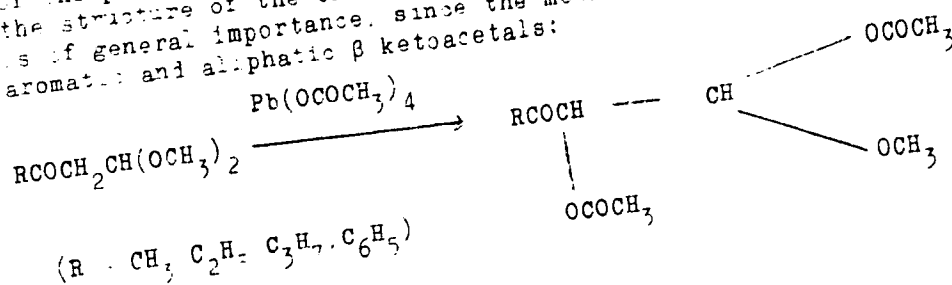
TEXT: The highly reactive and accessible β -ketoacetals $\text{ROOCH}_2\text{CH(OR')}_2$ (Refs. 1-4) are being used more and more in synthetic chemistry though some of their very promising reactions have scarcely been investigated up to now. The reactivity of the central methylene group has been given least consideration, particularly as far as the substitution of its hydrogen atoms is concerned (Refs. 5,6). The present publication deals with the oxidation of β -ketoacetals by means of lead tetraacetate.¹ Using this method, the authors (Ref. 3) and independently of them, other authors (Refs. 7,8) were able to introduce oxygen into the methylene group of β -ketoacetals.

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Oxidation of β -Ketoacetals by Means of Lead Tetraacetate

S/079/60/030/006/032/033/XX
B001/B055

but gave no details concerning the reaction. Recently however, when this investigation was practically concluded, a full description of the oxidation of acetaldehyde dimethyl acetal has been published in Ref. 9. The authors mainly studied the structure of the reaction products. Without giving direct evidence, the authors of Refs. 7 - 9 showed that the reaction proceeds according to Scheme 1. The authors of the present paper also found two acetoxy groups, which confirms the structure of the oxidation product. The oxidation of β -ketoacetals is of general importance, since the method can be applied to both aromatic and aliphatic β ketoacetals:



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Hydrogenolysis of Tetrahydrofurans

S/079/60/030/006/033/033/XX
B001/B055

are obtained which are not readily accessible by other methods, while in the second case only invaluable paraffin hydrocarbons are formed. There are 2 tables and 9 references: 4 Soviet 4 US and 1 British.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR
(Institute of Organic Chemistry of the Academy
of Sciences USSR)

SUBMITTED: June 29 1969

Carl S/3

S/079/60/030/007/034/039/XX
B001/B066

AUTHORS: Kochetkov, N. K., Nifant'yev, E. Ye., and Shibayev, V. N.

TITLE: Synthesis of Acyl-2-chloro-cyclohexenes-2 and Ethylene
Ketals of 2-Acyl-cyclohexanones. A New Synthesis of Phen-
anthrenes \uparrow

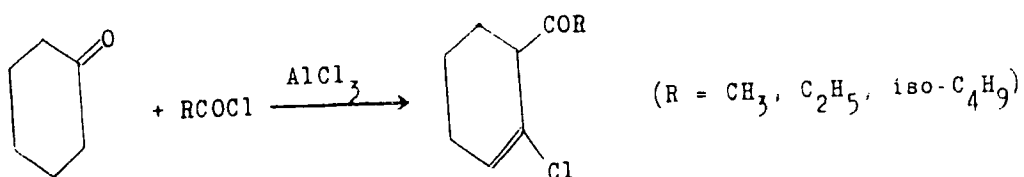
PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 7, pp. 2275-2282

TEXT: The authors describe the synthesis of the ethylene ketals of 2-acyl-cyclohexanones which have not been described as yet and were used as the starting material in a more convenient method of synthesizing phenanthrene derivatives. The synthesis was made on the basis of acyl-2-chlorocyclohexenes-2 which had been obtained by the authors in Ref. 1 by condensation of cyclohexanone with acid chlorides in the presence of $AlCl_3$, most suitably in a molar ratio of 2-3 $AlCl_3$: 2-3 acid chloride : 1 ketone: ✓

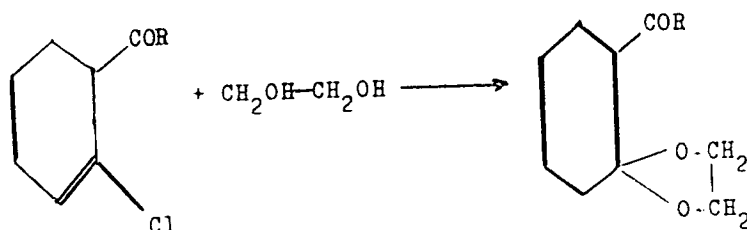
Card 1/4

Synthesis of Acyl-2-chloro-cyclohexenes-2 and Ethylene Ketals of 2-Acyl-cyclohexanones. A New Synthesis of Phenanthrenes

S/079/60/030/007/034/039/XX
B001/B066



The reaction must be carried out at low temperature since otherwise resinification occurs (yield, 45-80%). On reaction of acyl-2-chlorocyclohexene-2 with ethylene glycol which has been earlier used by the authors (Refs. 2, 10, 11), the ethylene ketals of 2-acyl-cyclohexanones were obtained (50-60%)



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Synthesis of Acyl-2-chloro-cyclohexenes-2 S/079/60/030/007/034/033/XX
and Ethylene Ketals of 2-Acyl-cyclo- B001/B066
hexanones. A New Synthesis of Phenanthrenes

The best solvent is dioxane. Ethylene ketals of 2-acyl-cyclohexanones in which one of the carbonyl groups is protected, are a convenient starting material. In this case, they were used as initial compounds for a new synthesis of the phenanthrene system. This synthesis is closely related to the synthesis of the naphthalene ring described by the authors in Refs. 10, 12, and is performed according to scheme 3. On reaction of the ethylene ketals with benzyl magnesium chloride, the corresponding oxy-ketals are formed which are directly converted to 1,2,3,4-tetrahydro-phenanthrenes by aromatic cyclodehydration. The best condensing agents were hydrogen bromide in acetic acid, or mixtures of concentrated sulfuric and phosphoric acid. Tetrahydrophenanthrenes are separable by distillation. They are purified by producing the picrates. By this method, some 10-alkyl-1,2,3,4-tetrahydrophenanthrenes hitherto unknown were obtained in yields of between 25 and 55%. The structure of the resultant compounds was confirmed by the absorption spectra in ultraviolet, which are characteristic of the tetrahydrophenanthrene ring. The resultant tetrahydro-phenanthrenes are quantitatively converted to 9-alkyl-phenanthrenes when heated with palladium-on-carbon (Scheme 4). There are 19 references: 10 Soviet, 5 US,

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Synthesis of Acyl-2-chloro-cyclohexenes-2
and Ethylene Ketals of 2-Acyl-cyclo-
hexanones. A New Synthesis of Phenanthrenes

S/079/60/030/007/034/039/XX
B001/B066 ✓

1 British, 2 German, and 2 French.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet
(Moscow State University)

SUBMITTED: July 6, 1959

Card 4/4

5.3400

67951

5(3)

AUTHORS:

Nifant'yev, E. Ye., Molodtsov, N. V., Kudryashov, L. I.,
Kochetkov, N. K.

SOV/20-130-1-26/69

TITLE:

Ethylene Acetals of α -Bromaroylacetaldehydes and Their
Transformations

PERIODICAL:

Doklady Akademii nauk SSSR, 1960, Vol 130, Nr 1, pp 94-97 (USSR)

ABSTRACT:

The authors wanted to synthesize β -ketoacetals with functional groups in the molecule. For this purpose, they investigated the exchange reaction of the bromine atom in the α -bromo- β -ketoacetals RCO-CHBr-CH(OR)_2 the synthesis method of which they had worked out recently (Ref 2). α -Bromo-substituted ethylene acetals of the aromatic series $\text{ArCOCHBrCH(OCH}_2)_2$ were best suited. Such compounds were produced by bromination of the ethylene acetals of aroylacetaldehydes (see Scheme). The bromination was achieved either by bromine action in ethereal solution in the presence of barium carbonate (Ref 2) or by bromosuccinimide. The products obtained and mentioned in the title are stable, crystalline substances. Their bromine atom is quite readily exchanged by interaction with salts of some mineral acids. Thus, corresponding α -substituted ethylene acetals of

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**Ethylene Acetals of α -Bromobenzoylacetaldehydes and
Their Transformations**

67951

SOV/20-130-1-26/69

aroylacetaldehydes (see Scheme) are formed, namely α -iodine- and α -thiocyanogen-substituted ethylene acetals. A little more difficult is the substitution of bromine by the nitro group while α -nitro- β -ketoacetal is formed. The above compounds represent a valuable initial material for the synthesis of some hardly accessible substances such as 4-benzoyl-2-oxythiazol. The interaction of brominated ketoacetals with mercaptanes proceeds smoothly. The reaction of the ethylene acetal of α -bromobenzoylacetaldehyde with sodiumbenzylmercaptide in methanol produces the ethylene acetal of α -benzylthiobenzoylacetaldehyde (see Scheme, Fig 1: I - the UV spectrum). The same bromoacetal reacts differently with sodium phenolate. No pure compound could be isolated from the resulting complex mixture by the reaction in acetone. On the other hand, the same reaction in methanol yielded a crystalline substance the analysis of which corresponded to the β -phenoxy- β -methoxy- α -oxy-hydrocinnamic aldehyde. Its UV spectrum (Fig 1: II) proves the missing benzoyl group and confirms the structure mentioned. It seems that the reaction with sodium phenolate proceeds via a transient α -oxide (similar to reactions described by T. I. Temnikova, Ref 5, see Scheme).

Card 2/3

Ethylene Acetals of α -Bromaroylacetaldehydes and
Their Transformations

67551
SOV/20-130-1-26/69

The interaction of bromoketosacetals with amines is complicated by the fact that - besides the exchange of the bromine atom - the acetal group enters the reaction. Thus, the phenyl- α,β -di-N-piperidylvinylketone develops in a high yield from the ethylene acetal of the α -bromobenzoylacetaldehyde and piperidine (UV spectrum, Fig 1: IV). Table 1 shows the constants and yields of the substances produced. There are 1 figure, 1 table, and 7 references, 5 of which are Soviet. ✓

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University imeni M. V. Lomonosov)

PRESENTED: June 9, 1959, by A. N. Nesmeyanov, Academician

SUBMITTED: June 6, 1959

Card 3/3

KOCHETKOV, M.K.; SOKOLOV, S.D.; VAGURTOVA, M.M.; NIFANT'YEV, E.Ye.

Organomagnesium compounds of the isoxazole series. Dokl.
AN SSSR 133 no.3:598-601 J1 '60. (MIRA 13:7)

1. Moskovskiy gosudarstvennyy universitet imeni M.V.
Lomonosova. Predstavleno akad. A.M.Mesmeyanovym.
(Magnesium organic compounds)
(Isoxazole)

KOCHETKOV, N.K.; NIFANT'YEV, E.Ye.

Chemistry of β -keto acetals. Usp. khim. 30 no. 1:31-47 Ja '61.
(MIRA 14:2)

1. Khimicheskiy fakul'tet Moskovskogo gosudarstvennogo
universiteta imeni M.V. Lomonosova.
(Acetals)

PETROV, K.A.; NIFANT'YEV, E.Ye.; NIKITINA, R.F.

Synthesis of diaryl phosphates and aryl phosphonates, and some
of their properties. Zhur.ob.khim. 31 no.5:1705-1709 My '61.
(Phosphoric acid) (Phosphonic acid) (MIRA 14:5)

PETROV, K.A.; NIFANT'YEV, E.Ye.; LYSENKO, T.N.

New synthesis of dialkyl phosphates. Zhur.ob.khim. 31 no.5:1709-
1711 My '61. (MIRA 14:5)

(Phosphoric acid)

PETROV, K.A.; NIFANT'YEV, E.Ye.; GOL'TSOVA, R.G.

Interesterification of methyl phosphonites. Zhur.ob.khim.
31 no.7:2367-2370 J1 '61. (MIRA 14:7)
(Phosphonic acid) (Esterification)

PETROV, K.A.; NIFANT'YEV, E.Ye.; GOL'TSOVA, R.G.

Interesterification of monoethyl methylphosphinite with glycols.
Zhur.ob.khim. 31 no.7:2370-2373 J1 '61. (MIRA 14:7)
(Phosphinic acid) (Esterification) (Glycols)

PETROV, K.A.; NIFANT'YEV, E.Ye.; LYSENKO, T.N.; YEVDKOV, V.P.

Synthesis of esters of phosphorous and phosphinic acids by
alcoholysis of their amides. Zhur.ob.khim. 31 no.7:2377-2380
Jl '61. (MIRA 14:7)

(Phosphorous acid) (Phosphinous acid)

NI FANT'YEV E Ye

25371

S/079/61/031/008/009/009
D215/D304

5.3630

AUTHORS: Petrov, K.A., Nifant'ev E.Ye., Gol'tsova, R.G.
and Gubin, G.V.

TITLE: Investigating the chemical properties of acid bis-
esters of ethylene glycol and methylphosphine acid

PERIODICAL: Zhurnal obshchey khimii, v. 31, no. 8, 1961, 2732-2735

TEXT: In previous publications, the authors have shown that acid bis-
methylphosphonites could be prepared by esterification of the mono-
ethylester of methylphosphinic acid with glycols. In the present in-
vestigations they studied some reactions of the simplest of these com-
pounds, obtained by esterification with ethylene glycol. The follow-
ing reactions were studied. 1) Oxidation of bismethylphosphinite
[Abstractor's note: Called subsequently "the starting product"] with
nitrogen oxides to the corresponding ester of bismethylphosphonic acid,
according to scheme (NI). The obtained product is highly hygroscopic
and reacts as a dibasic acid. 2) The reaction of the starting product

Card 1/2

25371

S/079/61/031/008/009/009
D215/D304

Investigating the chemical...

with sulfur; they did not succeed with the product itself, only with its sodium salt which was obtained from the product and sodium methoxide in dry methyl alcohol and could be isolated. (N2). 3) The reaction with dibutyldisulfide and methylthiocyanate (N3). 4) chlorination of the starting product which was successful with chlorine, but not with SO_2Cl_2 ; only a monochloride was obtained with chlorine which was oxidized to a corresponding phosphonic acid (N4). 5) Aminomethylation with tetraethyldiaminomethylene; with equimolar amounts of reagents they obtained monomethyl diethylamino phosphonate (N5). In the last two reactions the two phosphonic groups showed a different reactivity, only one of them taking part in the reaction. There are 5 references: 3 Soviet-bloc and 2 non-Soviet-bloc. The references to the English-language publications read as follows: L.W. Daasen, J.Am.Chem.Soc. 80,5301, 1958. E.K. Fields, J.Am. Chem.Soc. 74, 1528, 1952.

SUBMITTED: July 27, 1960

Card 2/2

S/079/61/031/009/001/012
D215/D306

AUTHORS: Petrov, K.A., Nifant'yev, E.Ye., and Khorkhoyanu, L.V.

TITLE: Peresterification of esters of dialkyl-phosphinous acids with glycerine derivatives

PERIODICAL: Zhurnal obshchey khimii, v. 31, no. 9, 1961,
2889 - 2894

TEXT: In the present work the authors studied peresterification of dialkyl- and diarylphosphinous acids with glycerine derivatives containing one free hydroxyl group for use in insect repellent compounds. The reaction of 1,2-diphenylideneglycerine with 1,2-isopropylideneglycerine was studied. The compounds were found to react readily with simpler esters, methylethyl-, dipropyl- and diphenylphosphinous acid. Glycerine derivatives with free secondary hydroxyls such as 1,3-benzylideneglycerine reacted less readily, but still gave good yields of the corresponding phosphinites. The phosphinites of the glycerine series provide novel compounds which

Card 1/6

Peresterification of esters ...

S/079/61/031/009/001/012
D215/D306

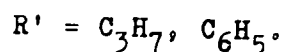
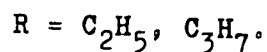
are either liquids or crystalline solids having unpleasant odours. They oxidize easily in air but remain stable in an inert gas atmosphere; their chemical properties are similar to those of simpler dialkyl- and diarylphosphinous acids and in oxidizing medium and in the presence of sulphur convert to the corresponding phosphonates and thiophosphonates. The synthesized phosphinites react according to Arbuzov's reaction forming phosphine oxides and corresponding halogen derivatives. The peresterification and alkylation of phosphinites may be used in preparing some halogen derivatives from polyatomic alcohols if the former are difficult to produce. In the present work the authors also investigated this reaction in order to produce more complex halogen derivatives of the polyatomic alcohols. The propyl dipropylphosphinite and ethyl diphenylphosphinite necessary for this reaction were prepared by reacting Menshutkin acid chlorides with organomagnesium compounds at -70°C



Card 2/6

Peresterification of esters ...

S/079/61/031/009/001/012
D215/D306



(1)

The first of the esters obtained has been unknown so far, and the second used to be prepared by more complex methods. The peresterification reaction was conducted by heating equimolecular quantities of the phosphinite and the glycerine derivative in a dry nitrogen stream with a small piece of sodium, distilling the required quantity of alcohol and finally vacuum distilling the residue. Time, temperature, yields and constants of the compounds obtained are given in tabulated form. In further experiments the propyl ester of dipropylphosphinous acid was oxidized with nitrogen oxides at $-100^{\circ}C$ until a permanent green coloration was obtained. Vacuum distillation of the solution yielded the propyl ester of dipropylphosphinic acid with high purity; b.pt. $103-104^{\circ}C/1$ mm Hg, $n_D^{20} - 1.4418$, $d_4^{20} - 0.9543$, and having an empirical formula

Card 3/6

Peresterification of esters ...

S/079/61/031/009/001/012
D215/D306

$C_9H_{21}O_2P$. The ester is colorless, odorless, insoluble in water, and soluble in ether, benzene, carbon tetrachloride and petroleum ether. Similarly oxidations of 1,2-isopropylidene glycerine ester and the 1,3-benzylideneglycerine ester of dipropylphosphinous acid were carried out to yield respectively esters of dipropylphosphinic acid, $C_{12}H_{25}O_4P$, b.pt. 143-144°/0.2 mm, n_D^{20} - 1.4530, d_4^{20} - 1.0376 and $C_{16}H_{21}O_4P$ b.pt. 117-118°/10⁻⁴ mm n_D^{20} - 1.5190. Both esters are insoluble in water and petroleum ether and soluble in alcohol, acetone, benzene, chloroform and carbon tetrachloride. Addition of sulphur to both propyl- and 1,2-isopropylideneglycerine esters of dipropylphosphinous acid was conducted by heating the esters with thoroughly dry sulphur at 140-142°C (exothermic reaction). The corresponding sulphur derivatives have b.pts. 81-82°C/0.5 mm and 141-140°C/1 mm respectively, unpleasant odors, and are both insoluble in water and soluble in

Card 4/6

Peresterification of esters ...

S/079/61/031/009/001/012
D215/D306

common organic solvents. Propyl-, 1,2-isopropylideneglycerine- and the 1,3-benzylideneglycerine esters of dipropylphosphinous acid undergo the Arbuzov rearrangement with methyl iodide to yield respectively dipropylmethylphosphine oxide, b.pt. 91-93°C/1 mm, m.pt. 39-39.5°C, the above oxide and 2,2-dimethyl-4-iodomethyldioxolen-1,3. b.pt. 81-83°C/9 mm. n_D^{20} - 1.5038, and the oxide as before and 2-phenyl-5-iodo-dioxan b.pt. 117-120°C/9 mm, n_D^{20} - 1.4983. The preparation of propyl ester of dipropylphosphinous acid and the ethyl ester of diphenylphosphinous acid was carried out by reacting the corresponding alkyl (propyl or ethyl) dichlorophosphite, pyridine, alcohol and propyl- or phenylmagnesium bromide respectively in ether at -65°C. Distillation of the reaction mixture yields in the first case the propyl ester of dipropylphosphinous acid b.pt. 70-71°C/7 mm, n_C^{20} - 1.4430, d_4^{20} - 0.8473 MR_{found} 54.64; MR_{calculated} 54.94, which is a mobile liquid with unpleasant smell which igni-

Card 5/6

Peresterification of esters ...

S/079/61/031/009/001/012
D215/D306

tes in air and which is insoluble in water but soluble in common organic solvents; in the second case the ethyl ester of diphenylphosphinous acid b.pt. 127-128°C/1 mm, n_D^{20} - 1.5910. There are 1 table and 8 references: 4 Soviet-bloc and 4 non-Soviet-bloc. The references to the English-language publications read as follows: U.S. Patent 2,588,407; E. Baer, H.L. Fischer, J. Am. Chem. Soc. 70, 609, 1948; C.N. Smith, D. Burnett, J. Econ. Entomol. 42, 434, 1949; T.H. Bevan, T. Malkin, D.B. Smith, J. Chem. Soc. 1955, 1383.

SUBMITTED: September 5, 1960

Card 6/6

PETROV, K.A.; NIFANT'YEV, E.Ye.; KHORKHOYANU, L.V.; TRUSHKOV, A.I.

Reesterification of esters of dialkyl- and diarylphosphinic acids.
Zhur.ob.khim. 31 no.9:3085-3090 S '61. (MIRA 14:9)
(Phosphinic acid) (Esterification)

PETROV, K.A.; NIFANT'YEV, E.Ye.; GOL'TSOVA, R.G.

Re-esterification of phosphinothioic and phosphonothioic esters
with alcohols. Zhur.ob.khim. 31 no.10:3174-3177 0 '61.
(MIRA 14:10)
(Phosphonothioic acid) (Phosphinothioic acid) (Alcohols)

NIFANT'YEV, E. YE.

43

PHASE I BOOK EXPLOITATION

SOV/6034

Konferentsiya po khimii i primeneniyu fosfororganicheskikh soyedineniy. 2d,
Kazan', 1959

Khimiya i primeneniye fosfororganicheskikh soyedineniy; trudy (Chemistry
and Use of Organophosphorus Compounds; Conference Transactions) Moscow,
Izd-vo AN SSSR, 1962. 630 p. Errata slip inserted. 2800 copies printed.

Sponsoring Agency: Akademiya nauk SSSR. Kazanskiy filial.

Resp. Ed.: A. Ye. Arbuzov, Academician; Ed. of Publishing House: L. S.
Povarov; Tech. Ed.: S. G. Tikhomirova.

PURPOSE: This collection of conference transactions is intended for chemists,
process engineers, physiologists, pharmacists, physicians, veterinarians,
and agricultural scientists.

COVERAGE: The transactions include the full texts of most of the scientific
papers presented at the Second Conference on the Chemistry and Use of

Card 1/14

43

Chemistry and the Use of Organophosphorus (Cont.)

SOV/6034

Organophosphorus Compounds held at Kazan' from 2 Nov through 1 Dec 1959. The material is divided into three sections: Chemistry, containing 67 articles; Physiological Activity of Organophosphorus Compounds, containing 26 articles; and Plant Protection, containing 12 articles. The reports reflect the strong interest of Soviet scientists in the chemistry and application of organophosphorus compounds. References accompany individual reports. Short summaries of some of the listed reports have been made and are given below.

TABLE OF CONTENTS. [Abridged]:

Introduction (Academician A. Ye. Arbuzov)

3

TRANSACTIONS OF THE CHEMISTRY SECTION

Gefter, Ye. L. [NII plastmass (Scientific Research Institute of Plastics, Moscow)]. Some Prospects for the Industrial Use of Organophosphorus Compounds

46

Card 2/14

Chemistry and the Use of Organophosphorus (Cont.)

SOV/6034

substituted phosphoric and phosphonic acids, as well as phosphorus-containing catalysts, have been synthesized and studied.

Petrov, K. A., V. A. Parshina, and G. L. Daruze. Phosphorus-Containing Polyester and Polyamide Resins

285

Bis-(p-carboxyphenyl)phosphonic acid, its esters, and salts, as well as amides and chlorides, have been obtained and for the first time described in the scientific literature. Organophosphorus polyesters and polyamides based on ethylene glycol, diethylene glycol, hexamethylenediamine, and bis-(p-carboxyphenyl)phosphonic acid and capable of forming fibers have been obtained and described.

Petrov, K. A., E. Ye. Nifant'ev, and I. I. Sopikova. Application of Arbuzov's Rearrangement to the Synthesis of Polyphosphonates

292

Previously unknown cyclic phenyl phosphonites have been obtained by the interaction of phenyldichlorophosphine with 1,3-glycol. The phosphonites polymerize under the action of methyl iodide,

Card 7/14

S/190/62/004/002/012/021
B:10/B'01

AUTHORS: Petrov, K. A., Nifant'yev, E. Ye.

TITLE: Phosphorylated polysaccharides. I. Phosphorylation of cellulose by transesterification of esters of acids of trivalent phosphorus

PERIODICAL: Vysokomolekulyarnyye soyedineniya, v. 4, no 2, 1962.
242 - 245

TEXT: Polysaccharides are phosphorylated by transesterification of esters of acids of trivalent phosphorus (neutral and acid phosphites and acid methyl phosphites). After 25-hr heating of diethyl phosphite together with anhydrous cellulose to 160 - 165°C in the presence of metallic sodium, the P content in the end product reaches only 2.1%, and under harder conditions 175 - 185°C, 6.4%. The phosphorylated polysaccharide dissolves in excess diethyl phosphite to a gelatinous substance, and is separated again by water addition. The optimum temperature of reaction with triphenyl phosphite is 100°C (P content 5 - 6%). In the reaction of cellulose with monomethyl ester (I) or monoethyl ester (II) of methyl phosphinous acid at 165 - 175°C and Na catalyst, the P content in the end product is 19.1%. One, two, or Card 1/2

Phosphorylated polysaccharides...

S/120,62/004, 002/011, 021
B110/B101

three ester alkoxyls may react with the OH groups of cellulose. Since 2.05 g of inulin are also phosphorylated by 8 g of II by 40 hr heating at 170-180°C (23% P; η > 200), the method is of universal importance. The esters look like cellulose, are relatively inseparable (cellulose phosphinite with η = 150 does not burn), are soluble in trimethyl benzyl ammonium hydroxide and warm H_3PO_4 , partly swelling in organics, and partly hydrolyzing on prolonged treatment with H_2O . Reactions reveal the presence of trivalent phosphorus. After prolonged standing oxidation to pentavalent, hitherto unknown derivatives occurs. There are 10 references: 1 Soviet and 9 non-Soviet. The four most recent references to English-language publications read as follows: K. Weller, Canad. Text. J., 70, 75, 1955; R. F. Schwenker, E. Pasck, Industr. and Engng. Chem., 50, 91, 1958; G. P. Toney, US Patent 2759924, Chem. Abstr., 51, 713, 1957; S. Hobart et al., Text. Res. J., 29, 884, 1959.

SUBMITTED: February 9, 1961

Card 2/2

15 8150

11.9700

11.2230

133382

S/190/62/004/002/013/021

B110/B101

AUTHORS: Petrov, K. A., Nifant'yev, E. Ye., Khorkhoyanu, L. V.,
Merkulova, M. I., Voblikov, V. F.

TITLE: Phosphorus-containing polymers. III. Application of the
Arbuzov reaction for polymerizing ethylene alkyl phosphites

PERIODICAL: Vysokomolekulyarnyye soyedineniya, v. 4, no. 2, 1962, 246-249

TEXT: The method by A. Ye. Arbuzov et al. (Izv. AN SSSR, Otd. khim. n., 1950, 357) can be used for producing polyphosphonates from cyclic phosphinites. In the present study, polyphosphonates were similarly synthesized on the basis of ethylene alkyl phosphites (I). Alcohol was added dropwise to 126.5 g of ethylene chlorophosphite, 300 ml of ether, and 152 g of triethylamine; the mixture was left standing, filtered off, heated for 30 min, and (I) was obtained by double distillation. Cyclic phosphites contain an alkoxy group besides the cyclic ester group. Polyphosphonates are formed under catalytic action of methyl iodide on ethylene alkyl phosphite during 3 hr heating at 130°C in Ar atmosphere: ✓

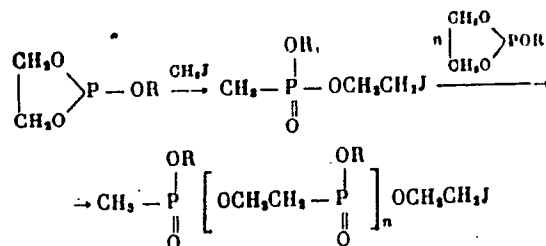
Card (1/3)

3382

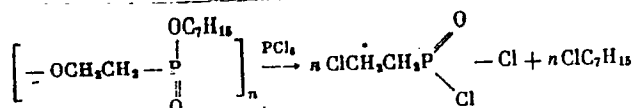
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B110/B101

Phosphorus-containing polymers...



The structure of polyethylene heptyl phosphite was proven as follows:

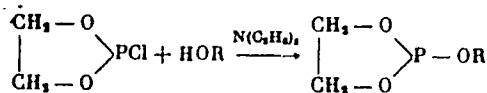


The degree of polymerization depends on the CH_3I amount, the reaction time and temperature. Optimum was: (1) small CH_3I amount; (2) ~20 - 30 hr, the reaction time depending on the molecular weight of the monomer,
Card 2/3

Phosphorus-containing polymers...

33382
S/190/62/004/002/013/021
B110/B101

the reaction temperature, and the CH_3I concentration; (3) $\sim 160 - 200^\circ\text{C}$, depending on the molecular weight (hexyl and isooctyl compounds: $160 - 170^\circ\text{C}$; nonyl and decyl compounds: 200°C). The polymers are viscous, colorless, and odorless liquids soluble in organics. Some of them are highly thermostable (polydecyl ethylene phosphite endures $\leq 200^\circ\text{C}$ for 20 - 30 hr). Utilization as plasticizer or admixture to lubricants is possible.



was also synthesized. There are 2 tables and 5 references: 4 Soviet and 1 non-Soviet. The reference to the English-language publication reads as follows: A. K. Sherrill, J. Amer. Chem. Soc., 52, 1985, 1930.

SUBMITTED: February 9, 1961

Card 3/3

PETROV, K.A.; YEVDKOV, V.P.; BILEVICH, K.A.; RADCHENKO, V.P.; NIFANT'YEV,
E.Ye.

Properties of phosphorus acid amides. Part 1: Reactions of
amidophosphites with phenols. Zhur.ob.khim. 32 no.3:920-
923 Mr '62. (MIRA 15:3)
(Phosphoramidous acid) (Phenols)

PETROV, K.A.; NIFANT'YEV, E.Ye.; SHCHEGOLEV, A.A.

Glucose phosphinites. Zhur.ob.khim. 32 no.3:1006 Mr '62.
(MIRA 15:3)
(Glucose) (Phosphinic acid)

PETROV, K.A.; NIFANT'YEV, E.Ye.; GOL'TSOVA, R.G.; BELAVENTSEV, M.A.;
KORNEYEV, S.M.

Esterification of phosphorous and phenylphosphinic acids. Zhur,-
ob.khim. 32 no.4:1277-1279 Ap '62. (MIRA 15'4)
(Phosphorous acid) (Phosphinic acid) (Esterification)

PETROV, K.A.; NIFANT'YEV, E.Ye.; SHCHEGOLEV, A.A.; KHUDYNTSEV, N.A.

Synthesis and chemical properties of phosphinites of 1,4;3,6-
dianhydrohexitol. Zhur.ob.khim. 32 no.9:3074-3080 S '62.
(MIRA 15:9)

(Hexitol) (Phosphinic acid)

43311
S/079/62/032/011/008/012
D204/D307

5 220
AUTHORS:

Petrov, K.A., Nifant'yev, E.Ye., and Gol'tsova, R.G.

TITLE:

Peresterification of phosphites and phosphinites with substituted alcohols

PERIODICAL:

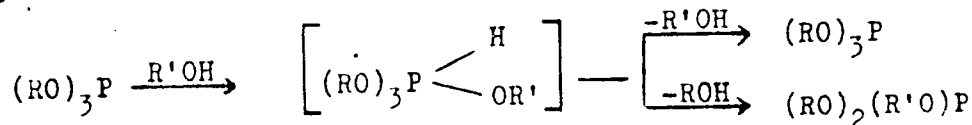
Zhurnal obshchey khimii, v. 32, no. 11, 1962,
3716 - 3720

TEXT: The peresterification of the simpler esters of phosphorous acid and methyl-, phenyl-, and dipropylphosphinous acids was studied, using amino-ethanol, halo- and cyanhydrins, furfuryl and tetrahydrofurfuryl alcohols and with methyl cellosolve since the literature concerning such reactions is very scarce. In a typical experiment a mixture of the ethyl ester of the phosphorous (or phosphinous) acid and the substituted alcohol was heated, under an inert atmosphere, to 150-185°C, until the calculated quantity of EtOH distilled off. The reaction mixture was held at that temperature for a further 10-15 min, at 20-40 mm Hg, and was then distilled to give the corresponding phosphite or phosphinite of the substituted alcohol. Na or H_3PO_4 were used as catalysts. Furfuryl-di-
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Peresterification of phosphites ...

S/079/62/032/011/008/012
D204/D307

propyl- and - cyanoethyldipropylphosphinites were quantitatively oxidized to the corresponding phosphonates with a current of dry O_2 . The rates and yields of the peresterifications were lower when the substituents in the alcohol were more electrophilic and when they were closer to the OH-carrying carbon atom. These data are in agreement with the mechanism showed earlier by the authors, i.e.:



Those initial phosphites and phosphinites which possessed more strongly electrophilic substituents reacted more readily with the alcohols. Thus di- β -chloroethylphosphite and di- β -fluoroethylphosphite were reacted with decyl alcohol, at respectively 140-150°C and 120-130°C, in the presence of H_3PO_3 , to give didecylphosphite in 80 and 85 % yields. There is 1 table.

SUBMITTED: December 14, 1961
Card 2/2

S/079/62/032/011/009/012
D204/D307

AUTHORS: Petrov, K.A., Nifant'yev, E.Ye., and Khorkhoyanu, L.V.

TITLE: Phosphorylation of glycerine and its derivatives by
alcoholysis of the amides of dialkylphosphinous acids.
A new method of directed replacement of a hydroxyl by
a cyano group

PERIODICAL: Zhurnal obshchey khimii, v. 32, no. 11, 1962,
3720 - 3723

TEXT: Interactions of the diethylamide of dipropylphosphinous
acid (I) with 1,2-iso-propylideneglycerine (II), 1,3-benzylidenegly-
cerine (III) and glycerine were studied, in continuation of earlier
work (ZhOKh, 31, 2889, 1961). I and II, and I and III interacted
readily at 120-125°C to yield respectively the dipropylphosphini-
tes of 1,2-iso-propylideneglycerine and 1,3-benzylideneglycerine
(IV and V), in almost quantitative yields. Glycerine reacted analo-
gously, at 135-140°C, in 60 % yield, to give the corresponding tris-
dipropylphosphinite (VI). $C_3H_7OP(OC_3H_7)_2$ reacted readily with bu-

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Phosphorylation of glycerine and ...

S/079/62/032/011/009/012
D204/D307

tyl thiocyanate, at 0°C, under an inert atmosphere; when the exothermic reaction was over the mixture was heated at 100-110°C for 1 hr., and was then distilled to give BuSP(O)(Pr)₂. Cyanodesoxy-1,2-iso-propylideneglycerine (VII) was prepared from IV and benzyl thiocyanate, in 45 % yield, by an analogous reaction. VII was converted into iso-propylidene-2-desoxytetrose by mixing it into SnCl₂/ether/HCl, stirring for 1 hr. at the b.p. of the ether evaporating the latter off, adding water and neutralizing the mixture and heating for 5 hrs. at 50°C. Iso-propylidene-3,4-desoxy-4-aminoerythrite was obtained by adding VII to ethereal LiAlH₄ evaporating the ether off, adding an equivalent amount of THF and heating for 96 hrs. on a water-bath.

SUBMITTED: December 14, 1961

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43312
8/079/62/032/011/010/012
D204/D307

6 3630
AUTHORS:

Petrov, K.A., Nifant'yev, E.Ye., Goltsova, R.G.,
Shchegolev, A.A., and Bushmin, B.V.

TITLE:

Synthesis and peresterification of diphenyl phosphite

PERIODICAL:

Zhurnal obshchey khimii, v. 32, no. 11, 1962,
3723 - 3727

TEXT: The interactions of diphenyl phosphite with aliphatic alcohols were studied since the alcoholysis of diethyl and other simple phosphites (to higher phosphites) and phosphinites requires, in some cases, inconveniently high temperatures (this journal, p. 3716). Dialkyl phosphites $(RO)_2POH$, where $R = C_4H_9$, iso- C_5H_{11} , C_6H_{13} , C_8H_{17} , C_9H_{19} , $C_{10}H_{21}$, $ClCH_2CH_2$, and $C_2H_5OC(O)CH_2$, were prepared in 91-96 % yields by adding 2 moles ROH to 1 mole $(PhO)_2POH$ and heating for 3-8 hours at $100^\circ C$, in the presence or absence of catalyst (Na). The high reactivity of diphenyl phosphite as compared to those of simple dialkyl phosphites is ascribed to (1) the existence
Card 1/2

S/079/62/032/011/010/012
D204/D307

Synthesis and peresterification ...

of transitory forms $\left[\begin{array}{c} \text{PhO} \diagdown \\ \text{P} \begin{array}{l} \nearrow \text{OR} \\ \text{H} \\ \nearrow \text{OH} \end{array} \\ \text{PhO} \diagup \end{array} \right]$ and $\left[\begin{array}{c} \text{PhO} \diagdown \\ \text{P} \begin{array}{l} \nearrow \text{OR} \\ \text{H} \\ \nearrow \text{OH} \end{array} \\ \text{RO} \diagup \end{array} \right]$, which prefe-

rentially eliminate PhOH rather than ROH, owing to the considerably higher electrophilic character of the PhO group, and (2) the fact that the tautomeric equilibrium favors the trivalent P form far more in diphenyl than, say, in diethyl phosphite. Similar reactions took place readily with substituted alcohols such as e.g. $(\text{CH}_3)_2$

$\text{NCH}_2\text{CH}_2\text{OH}$. Diphenyl phosphite was obtained almost quantitatively by the equimolar interaction of diphenyl chlorophosphite with methanol (sealed tube, 100°C , 3 hrs.) and by the interaction of methyl dichlorophosphite with phenol (1:2) at 100°C for 1 hr. The latter method, which is generally convenient for the preparation of diaryl phosphites, was also used to make di-p- and di-m-cresyl phosphites, in ~100 % yields, by reacting CH_3OPCl_2 with para- and meta-cresols

There is 1 table.

SUBMITTED: December 14, 1961
Card 2/2

ACCESSION NR: AT4033987

S/0000/63/000/000/0068/0072

AUTHOR: Petrov, K. A.; Nifant'yev, E. Ye.; Gol'tsova, R. G.; Kornayev, S. M.

TITLE: Polymers containing phosphorus. IX. Synthesis of acid polyalkylene phosphites, phosphates and thionphosphates

SOURCE: Geterotsepnnyye vy*sokomolekulyarnyye soyedineniya (Heterochain macro-molecular compounds); sbornik statey. Moscow, Izd-vo "Nauka," 1963, 68-72

TOPIC TAGS: polymer, phosphorus containing polymer, polyalkylene phosphite, poly-alkylene phosphate, polyalkylene thionphosphate, linear acid polyphosphite, poly-phosphite synthesis, spatially discreet glycol, polyphosphite oxidation, poly-phosphite alkylation

ABSTRACT: Linear acid polyphosphites were synthesized by reesterification of di-ethyl phosphite with spatially discreet glycols, then converted to polyalkylene phosphates by NO_2 oxidation or to thionphosphates by reaction with S. Successful syntheses (procedure described) were obtained with pentandiol-1,5, hexandiol-1,6, diethylene glycol, triethylene glycol, diethanolamine, pentafluoropentandiol-1,5, 1,4-3,6-dianhydrosorbitol, and p-dihydroxymethylbenzene. A neutral polythion-phosphite was obtained by alkylation of an ammonium salt of polyalkylenethionphos-phoric acid. "We would like to thank S. A. Pavlova, associate at the INEOS AN SSSR, Card 1/2

ASSOCIATION: AT4033987

for her help in determining the molecular weights." Orig. art. has: 2 graphs, 1 table and 3 chemical equations.

ASSOCIATION: none

SUBMITTED: 19Jun62

DATE ACQ: 30Apr64

ENCL: 00

SUB CODE: OC

NO REF \$OV: 012

OTHER: 003

Card 2/2

ACCESSION NR: AT4017411

S/0000/63/000/000/0086/0089

AUTHOR: Petrov, K. A.; Nifant'yev, E. Ye.; Sopikova, I. I.; Merkulova, M. I.

TITLE: Phosphorylated polysaccharides. III. Phosphorylation of cellulose by dialkyl-(aryl)phosphites

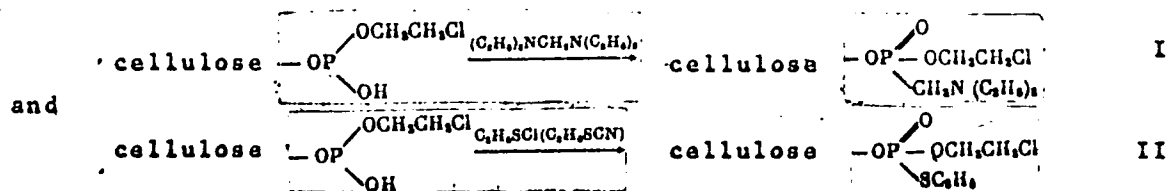
SOURCE: Tsellyuloza i yeye proizvodnyye, sbornik statey (Cellulose and its derivatives). Moscow, 1963, 86-89

TOPIC TAGS: polysaccharide, cellulose, phosphorylated polysaccharide, cellulose phosphorylation, phosphorylation, dialkylphosphite, diarylphosphite

ABSTRACT: On the basis of the authors' previous work, the following studies were conducted: (1) phosphorylation of cellulose by di- β -chloroethylphosphite, di- β -fluoroethylphosphite, and diphenylphosphite; (2) reaction of cellulose phosphite with tetraethylmethylenediamine; and (3) reactions of cellulose phosphite with chloral, diethyldisulfide, ethylsulfenechloride, and ethylthiocyanate. In the phosphorylation, 0.5 g of cellulose (cotton wool, thread and cord), dehydrated by washing with absolute alcohol, was reacted at 110, 130, 150 or 165C for 30 or 60 hrs. with 25 g of the reagents in a stream of nitrogen at a pressure of 50-60 mm Hg. After removal of the excess phosphite by filtration, the product was washed with methanol and ether and vacuum-dried over P_2O_5 . The A P and Cl content of 4.27-
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ACCESSION NR: AT4017411

8.56% and 2.97—9.06%, respectively, was found in the product, obtained from cotton wool, while lower results were achieved with viscose thread and cord. Di-8-chloro- and di-8-fluoroethylphosphites were found to be better suited for the reaction. In the reaction with tetra-ethylmethylenediamine and disulfides, conversion of cellulose phosphites into α -hydroxy- and α -aminophosphonates and thiolophosphates was also accomplished by the reactions



Orig. art. has: 1 table.

ASSOCIATION: none

Card 2/3

ACCESSION NR: AT4017411

SUBMITTED: 12Apr62

ATD PRESS: 3045

ENCL: 00

SUB CODE: OC

NO REF SOV: 006

OTHER: 002

Card 3/3

ACCESSION NR: AT4017412

S/0000/63/000/000/0090/0093

AUTHOR: Petrov, K. A.; Nifant'yev, E. Ye.; Sopikova, I. I.; Belavintsev, M. A.

TITLE: Phosphorylated polysaccharides. IV. A method for phosphorylating cellulose using phosphorous acid

SOURCE: Tsellyuloza i yeye proizvodny*ye, sbornik statey (Cellulose and its derivatives). Moscow, 1963, 90-93

TOPIC TAGS: polysaccharide, polysaccharide phosphorylation, cellulose, cellulose phosphate, phosphocellulose, phosphorylation

ABSTRACT: Cellulose was phosphorylated by phosphorous acid using 3 different procedures: (1) reacting cellulose and molten phosphorous acid at 100C for 10 hours in a current of dry nitrogen, yielding a product containing 15-17% P; (2) in dimethylformamide or o-xylene solutions in a series of 20 to 60-hour tests at 130 and 160C yielding a product containing 4.8-12.2% P; (3) prolonged (2-3 days) heating at 80-140C in an atmosphere of an inert gas, which proved to be the most suitable since it yielded products containing up to 14% P. Different kinds of cellulose were tested, and the one swollen in water or pyridine was found best. The P-content in the product increased with the concentration of phosphorous acid up to a certain limit, the optimal ratio being one in which there is slightly more than

Card 1/2

ACCESSION NR: AT4017412

one phosphorous acid molecule for each ¹³ C, d-glucose unit in the reacting mixture.
Orig. art. has: 2 graphs and 1 table.

ASSOCIATION: none

SUBMITTED: 12Jul62

DATE ACQ: 06Jan64

ENCL: 00

SUB CODE: CH

NO REF SOV: 006

OTHER: 003

Card 2/2

ACCESSION NR: AT4034002

S/0000/63/000/000/0170/0174

AUTHOR: Petrov, K. A.; Nifant'yev, E. Ya.; Gol'tsova, R. G.

TITLE: Phosphorus-containing polymers. X. Synthesis of polyphosphite-based polyphosphonates

SOURCE: Geterotsepnny*ye vy*sokomolekulyarny*ye soyedineniya (Heterochain macromolecular compounds); sbornik statey. Moscow, Izd-vo "Nauka," 1963, 170-174

TOPIC TAGS: polymerization, phosphorus containing polymer, polyphosphite, polyphosphonate

ABSTRACT: As a further step in the authors' polymer studies data are given on the synthesis of polyalkylalkylenephosphonates, poly- Δ -hydroxyalkylalkylene-phosphonates and poly- Δ -aminoalkylalkylenephosphonates by the Michaelis and Becker method using acid polyalkylenephosphites. The following polymers were prepared, identified and described: polybenzylhexamethylenephosphonate, poly- Δ -hydroxybenzylhexamethylenephosphate, poly- Δ -dibutylaminobenzylhexamethyl-enephosphonate, polydiethylaminomethylhexamethylenephosphonate, poly- Δ -propyl-aminoisopropylhexamethylenephosphonate, poly-diethylaminomethyl-p-xylylenephos-phonate, polybutylamino-bis-methylhexamethylenephosphonate, and a copolymer of
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ACCESSION NR: AT4034002

Δ -dibutylaminobenzylhexamethylenephosphonate and di-(hexamethylenephosphato) disulfide. The preparative procedure consists essentially of reacting the reagents for several hours at 90-135C; the yield varied from 48 to 98% for different individual products. Orig. art. has: 4 chemical equations.

ASSOCIATION: None

SUBMITTED: 13Nov62

DATE ACQ: 30Apr64

ENCL: 00

SUB CODE: OC

NO REF SOV: 009

OTHER: 004

Card 2/2

ACCESSION NR: AT4034009

8/0000/63/000/000/0240/0242

AUTHOR: Petrov, K. A.; Nifant'yev, E. Ya.; Ly*senko, T. N.

TITLE: Phosphorus-containing polymers. XI. Synthesis of hydrolytically stable polymers based on α -propylglucoside and N-phenylglucoside

SOURCE: Geterotsepnny*ye yy*sokomolekulyarny*ye soyedineniya (Heterochain macromolecular compounds); sbornik statey. Moscow, Izd-vo "Nauka," 1963, 240-242

TOPIC TAGS: polymerization, polymer, phosphorus containing polymer, alpha propylglucoside, phenylglucoside, polyphosphite, polyphosphinite

ABSTRACT: In an extension of the authors' previous work on phosphorus-containing polymers, a number of polyphosphites and polyphosphinites were obtained by the alcoholysis of phosphoamides and reesterification of arylphosphites and arylphosphinites, using α -propylglucoside and N-phenylglucoside as the reagents. In the alcoholysis procedure, 1 mol of α -methyl, α -propyl or N-phenylglucoside and 2 or 2.5 mols of phosphoamide ($C_3H_7OP[N(C_2H_5)_2]_2$, $C_4H_9OP[N(C_2H_5)_2]_2$, $C_8H_{17}OP[N(C_2H_5)_2]_2$) were heated at 140-145°C for 3 hrs., at 140-150°C/10 mm for 4 hrs. and at 180-190°C/3 mm for 3 hrs. in a stream of inert Card 1/2

ACCESSION NR: AT4034009

gas. Oxidation of the polyphosphites and polyphosphinites to polyphosphates and polyphosphonates with nitrogen dioxide was also conducted and the reaction of acid poly-N-phenylglucophosphite with chloral demonstrated. The polyglycophosphites and polyglycophosphinites obtained contain hydrophobic radicals and less thermo- and hydrolytically stable than the corresponding polyglycophosphates and polyglycophosphonates. Orig. art. has: 1 figure and 1 table.

ASSOCIATION: None

SUBMITTED: 24Apr63

DATE ACQ: 30Apr64

ENCL: 00

SUB CODE: OC

NO REF SOV: 003

OTHER: 003

Card 2/2

S/190/63/005/003/009/024
B101/B186

AUTHORS: Petrov, K. A., Nifant'ev, E. Ye., Khorkhoyanu, L. V.,
Voblikov, V. F.

TITLE: Phosphorylated polysaccharides. II. Phosphorylation of
cellulose by alcoholysis of amides of the acids of three-
valent phosphorus

PERIODICAL: Vysokomolekulyarnyye soyedineniya, v. 5, no. 3, 1963, 348-352

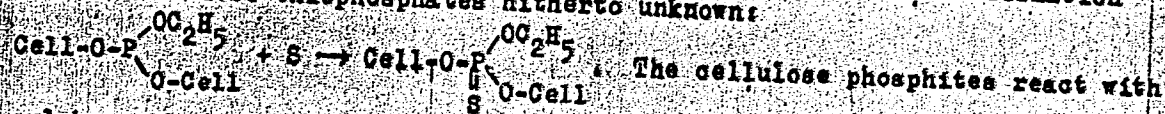
TEXT: In a previous paper (Zh. obshch. khimii, 31, 2377, 1961) the authors described the reaction: $\text{Cell-OH} + \text{R}_2\text{N}-\text{P} \rightarrow \text{Cell-O-P} + \text{R}_2\text{NH}$. In the present paper a study was made of this new method of phosphorylating cellulose by alcoholysis of phosphorous acid amides such as diethyl phosphorous acid diethylamide, ethylphosphorous acid tetraethylamide and phosphorous acid hexaethyltriamide in order to develop fireproof, antiseptic and insecticidal cellulose. Since the process of esterification of cellulose depends to a great extent on how the sample is prepared the following cellulose types were subjected to phosphorylation: viscose fiber, washed with methanol and dried; specially prepared cotton cellulose; cellulose

Card 1/3

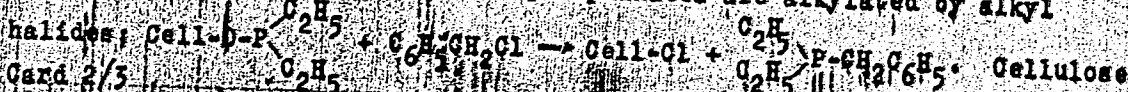
Phosphorylated polysaccharides...

S/190/63/005/003/009/024
B101/B186

reprecipitated from triethylbenzylammonium hydroxide; and pyridine-enclosed cellulose. The degree of phosphorylation increased with increasing temperature (80 - 120°C). Celluloses with a phosphorus content up to 17.4%, $\eta = 300$ were obtained. These optimum values were obtained with pyridine cellulose reprecipitated from triethylbenzylammonium hydroxide. With di- and triamides, insoluble compounds arose containing about 3 - 3.5% nitrogen so that cross linking is assumed. With monoamides, cellulose phosphinites were formed, soluble in methanol. The phosphinites with 5 - 7% P were extinguished again when the flame was removed, the esters with a still higher P content did not burn but only carbonized. The cellulose esters of the trivalent phosphorus acids are highly reactive. With dry oxygen quantitative oxidation to phosphates sets in. Sulfur adds with formation of the cellulose thiophosphates hitherto unknown:



sulphenechlorides. The cellulose-phosphinites are alkylated by alkyl



Card 2/3

Phosphorylated polysaccharides...

S/190/63/005/003/009/024
E101/E186

halides arise which can be used as the initial substances for the synthesis of desoxy-, cyano-, amino-, and other cellulose derivatives. There are 3 tables.

SUBMITTED: August 8, 1961

Card 3/3

I. 13552-63

ACCESSION NR: AP3000699

BNP(j)/KPP(c)/BWT(m)/BDS ASD PcaLi/Pr-Li RM/WW

8/0190/63/005/005/0712/0718

AUTHOR: Petrov, K. A.; Nifant'yev, E. Ye.; Ly*senko, T. N.; Suzanskiy, A. I. 64

TITLE: Phosphorus-containing polymers. 6. Synthesis of polyphosphites and polyphosphinites on the basis of glucose

SOURCE: Vy*sokmolekulyarny*ye soyedineniya, v. 5, no. 5, 1963, 712-718

TOPIC TAGS: polyphosphites, polyphosphinites, methylglucoside, phosphorylation, polymers, monosaccharides, polythiophosphates

ABSTRACT: The objective of the present investigation was an attempt to synthesize polymers, using methylglucoside from the hydrolysis of wood pulp and di- and triamides of trivalent phosphorous acids as the issuing materials. However, the alcoholysis by methylglucoside of tetraethylidiamides of methylphosphinic and ethylphosphorous acid and of hexaethyltriamide of phosphorous acid, when conducted at 100 to 130C, yielded bicyclic phosphinites with a molecular weight of only 210 to 220. Subsequent heating at 160 to 200C caused a molecular weight increase, with optimal molecular weights reaching 321.000 and 528.000, where the ratios of the reacting ingredients are close to equinormal. Since the formation of intermolecular bonds generally proceeds at a lesser rate when compared with the building of intracyclic phosphinite groups, it is necessary to conduct the polymerization in two steps, the

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L 13552-63

ACCESSION NR: AP3000699

second one intended to promote the conversion of the initially formed cyclic esters into branched polyphosphites and polyphosphinites. The obtained polymers could be converted to polyphosphates, polythiophosphates, and polyphosphonates by treatment with NO at 30 to 40C, with S at 130C, and with Arbuzov's alkylation reagent, respectively. Orig. art. has: 2 formulas, 3 figures, and 2 tables.

ASSOCIATION: none

SUBMITTED: 01Nov61

DATE ACQ: 17Jun63

ENCL: 00

SUB CODE: CH

NO REF SOV: 008

OTHER: 004

Card 2/2

PETROV, K.A.; NIFANTYEV, E.Ye.; GOZ'ISOVA, R.G.; SOLNTSEVA, L.M.

Phosphorus-containing polymers. Part 7: Synthesis of polyphosphites and polyphosphinites by glycolysis of amides of trivalent phosphorus acids. Vysokom.sped. 5 no.11:1691-1695 N '63. (MIRA 17:1)

PETROV, K.A.; NIFANT'YEV, E.Ye.; KHORKHOYANU, L.V.; GOL'TSOVA R.G.

Phosphorus-containing polymers. Part 8: Synthesis and some
properties of polyarylene phosphites and phosphinites.
Vysokom. soed. 5 no.12:1799-1804 D '63. (MIRA 17:1)

PETROV, K.A.; NIFANTSEV, E.Ye.; SHCHEGOLEV, A.A.

Synthesis of 1,2-dialkyl phosphinites; 5-6-diisopropylideneglucoses
and their conversion to 6-halodeoxyglucose. Zhur.ob.khim.
33 no.3:896-899 Mr '63. (MIRA 16:3)

(Phosphinic acid)
(Glucose)

PETROV, K.A.; NIFANT'YEV, E.Ye.; SHCHEGOLEV, A.A.; BUTILOV, M.M.; REBUS, I.F.

Re-esterification of neutral phosphites and phosphinites.

Zhur.ob.khim. 33 no.3:899-901 Mr '63. (MIRA 16:3)

(Phosphinic acid) (Phosphorous acid)

(Esterification)

PETROV, K.A.; NIFANT'YEV, E.Ye.; GOL'TSOVA, R.G.

Trans-esterification of diethyl phosphite with ethylene glycol.
Zhur. ob. khim. 33 no.5:1485-1488 My '63. (MIRA 16:6)

(Ethyl phosphites) (Esterification)
(Ethylene glycol)

NIFANT'YEV, E.Ye.; GRACHEV, M.A.; BAKINOVSKIY, L.V.; KARA-MURZA, S.G.;
KOCHETKOV, N.K.

Synthesis of methyl β -chlorovinyl ketone. Zhur.prikl.khim. 36 no.3:
676-678 My '63. (MIRA 16:5)
(Ketone) (Vinyl compounds)

PETROV, K.A.; NIFANT'YEV, E.Ye.; LIBMAN, B.Ya.

Synthesis of di-(2-ethylhexyl) phosphate and phenyldi-
(2-ethylhexyl) phosphate. Zhur. prikl. khim. 36 no.8:1853-
1857 Ag '83. (MIRA 16:11)

L 17550-63

EWP(j)/EPP(c)/EWT(m)/BDS Pc-4/Pr-4 RM/WW

ACCESSION NR: AP3004425

8/0020/63/151/004/0859/0861

AUTHORS: Petrov, K. A.; Nifant'ev, E. Ye.; Sopikova, I. I.

TITLE: Phosphorylation with acylphosphites.

SOURCE: AN SSSR. Doklady*, v. 151, no. 4, 1963, 859-861

TOPIC TAGS: phosphorylation, acylphosphite, alcohol, acid phosphonate

ABSTRACT: The purpose of this work was to develop new methods for phosphorylating hydroxyl compounds. Tribenzoylphosphite and butylene-1,3-acetylphosphite were used at relatively low temperatures in the presence of triethylamine. Tertiary phosphites were formed in high yields with primary, secondary, and tertiary alcohols and with acid phosphonates such as the monopropyl ester of methylphosphinic acid. They can also be used for phosphorylating carbohydrates and other natural products. The reaction of these acylphosphites with phosphoxanthogenates produced thiophosphates and other sulfur-containing compounds. These phosphorylating agents can be obtained readily by reacting PCl_3 or butylene-1,3-chlorophosphite with metallic salts of the corresponding carboxylic acid. Phosphoxanthogenates were produced by reacting PCl_3 and chlorophosphines with salts of alkylxanthogenic acids. Syntheses of the following are described: tributylphosphite; 1,3-butylenebutylphosphite; 1,3-butyleneisopropylphosphite; 1,3-butylene-tert.butylphosphite; Card 1/2

L 17550-63

ACCESSION NR: AP3004425

and O,O-1,3-butylene-O-propylmethylsubphosphonate. The preparation of tri-Alpha-furoylphosphite is also described. The original article has 3 formulas.

ASSOCIATION: none

SUBMITTED: 26Jan63

DATE ACQ: 21Aug63

ENCL: 00

SUB CODE: CH

NO REF SOV: 008

OTHER: 004

Card 2/2

POPOV, G.N.; NIFONTOV, B.I.; LOBANOV, D.P.; KULIKOV, A.V.;
KALYUZHNAIA, T.P., red.

[Characteristics of the development of radioactive ore
deposits] Osobennosti razrabotki mestorozhdenii radio-
aktivnykh rud. Moskva, Atomizdat, 1964. 218 p.
(MIRA 17:6)

L 34919-65

ACCESSION NR: AT5006104

S/0000/64/000/000/0042/0061

AUTHOR: Yerokhin, R. A.; Koshurnikova, N. A.; Lyubchanskiy, E. R.; Nifatov, A. P.; Reshetov, G. N. 10 13

TITLE: Content and microdistribution of plutonium-239 in rat lung and liver and morphological changes in these organs after intratracheal administration of the isotope

SOURCE: Raspredeleniye, biologicheskoye deystviye, uskoreniye vyvedeniya radioaktivnykh izotopov (Distribution, biological effect, acceleration of the excretion of radioactive isotopes); sbornik rabot. Moscow, Izd-vo Meditsina, 1964, 42-61

TOPIC TAGS: plutonium-239, radioisotope, inhalation, liver, lung, pathology, radioactivity, lymphatic system

ABSTRACT: The behavior of plutonium in the lung following intratracheal administration of various salts is determined largely by the physicochemical form of the compound used. The plutonium content of the lungs after administration of the nitrate was 5-10 times higher than after administration of sodium plutonyl triacetate. The clearance of plutonium administered in the form of these two salts obeys the exponential law, but it was more rapid in the case of the second salt. A large quan-

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L 34919-65

ACCESSION NR: AT5006104

tity of plutonium was transported from the lungs by macrophages into the regional lymph nodes. Plutonium accumulated in the liver during the early phase (20 minutes to 24 hours) more slowly after administration of the nitrate than it did after administration of sodium plutonyl triacetate. During the later phases (4 to 6 months) the rate of deposition in the liver was about the same after administration of either form of plutonium - 0.90-0.55 and 0.95-0.57% of the dose administered.

The microdistribution of plutonium in rat liver after intratracheal administration of the two plutonium salts was quite diffuse. Histological changes in the lung varied with the nature of the microdistribution of the element and they arose mainly in the places where the isotope concentrated. The severity of the pathological changes and the time when they developed were related to the ionization dose that accumulated. Among the earliest changes were degeneration, desquamation of bronchial and alveolar epithelium, and perivascular edema. These were followed by chronic inflammation, chiefly productive in character. The pathological process developed into pneumosclerosis as a result of the proliferation of connective-tissue cellular elements with the formation of fibrous structures. No significant morphological changes were noted in the liver after intratracheal administration of 7 μ c/kg of plutonium nitrate or sodium plutonyl triacetate. Orig. art. has: 15 figures, 2 tables.

Card 2/3

L 31919-65
ACCESSION NR: AT5006104

ASSOCIATION: none

SUBMITTED: 10Apr64

ENCL: 00

SUB CODE: LS

NO REF SOV: 000

OTHER: 000

Cord 3/3

L 34113-55 EWG(1)/EWI(m) GS

ACCESSION NR: AT5006122

S/0000/64/000/090/0183/0191

AUTHOR: Nifatov, A. P.; Buldakov, L. A.

TITLE: Biological effects of strontium-90, cesium-137, and promethium-147 after chronic oral administration of low doses of the isotopes 18
B+1

SOURCE: Raspredeleye, biologicheskoye deystviye, uskoreniye vyvedeniya radio-aktivnykh izotopov (Distribution, biological effect, acceleration of the excretion of radioactive isotopes); sbornik rabot. Moscow, Izd-vo Meditsina, 1964, 183-191

TOPIC TAGS: strontium-90, cesium-137, promethium-147, radioisotope, radioactivity, blood, viscera

ABSTRACT: Oral administration of 0.00051 and 0.012 $\mu\text{c}/24$ hours of Sr^{90} , 0.00124 $\mu\text{c}/24$ hours of Cs^{137} , and 0.0027 $\mu\text{c}/24$ hours of Pm^{147} to rats for almost two years had no significant effect on the average survival time, weight, peripheral blood, or rate of tumor formation in the rats. Although the overall number of tumors was the same in both the experimental animals and the controls, the spectrum of neoplasms was different. The experimental animals had more tumors of the adrenal cortex, testes, gastrointestinal tract, and generalized leukemias, a phenomenon not explained by the authors. Since the greater frequency of glandular tumors was not

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L 34113-65

ACCESSION NR: AT5006122

related to the dose or to local irradiation, the authors doubt that these neoplasms were the result of direct radiation injury or that they were the determining factor in the death of the animals. The main causes of death seemed to be various inflammatory processes in the lungs and gastrointestinal tract, periarteritis nodosa, cirrhosis of the liver, and nephrosclerosis. These diseases were equally common in the experimental and control animals. Orig. art. has 6 tables.

ASSOCIATION: none

SUBMITTED: 10Apr64

ENCL: .00

SUB CODE: LS

NO REF SOV: 000

OTHER: 000

Cord 2/2

L 23040-65 EPF(c)/EMP(f)/EMT(m) Pc-4/Pr-4 PM

ACCESSION NR: AP4044081

S/0189/64/000/004/0090/0090

AUTHORS: Nifant'yev, E.Ye.; Fedorov, S. G.

TITLE Phosphites and phosphonites of novolac resins. 15

SOURCE: Moscow. Universitet. Vestnik. Seriya 2. Khimiya, no. 4, 1964, 90

TOPIC TAGS: novolac phosphite, novolac /phosphonite, novolac resin, phosphorylation, intermolecular transphosphorylation, polyphosphite, polyamidophosphite, polyphosphite oxidation, polyphosphate, poly-oxyalkylphosphonate, aminomethylphosphonate, curing, urotropine

ABSTRACT: Novolac resins were phosphorylated with esters and amides of phosphorous and phosphonic acids. A novolac (molecular weight 630) reacted quantitatively at 150-170C with a three-fold excess of the monoethyl ester of methylphosphonic acid to form a phosphorylated resin I. Similar reaction at 160-170C with dimethylphosphite gave an organic solvent-soluble resin II containing segments of arylmethylphosphite. On heating above 170C II underwent intermolecular transphosphorylation to form an insoluble vitreous polymer, which may also be formed by conducting the phosphorylation reaction at 180-190C.

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L 23040-65

ACCESSION NR: AP4044081

Phosphorylation of the novolac at 140-150C with di- β -chlorethylphosphite gave polyphosphites which may, or may not, be cross-linked. The hexaethyltriamide of phosphorous acid reacted with novolac at 120-140C to form a soluble polyamidophosphite which was transformed to a three-dimensional polyphosphite on prolonged standing in vacuum. The polyphosphites and polyphosphonites formed were very reactive. II was oxidized to the polyphosphate. I and II reacted with aldehydes and tetraethyldiaminomethylene to form poly- α -oxyalkyl- and α -aminomethylphosphonates. Liquid I and II resins were cured with urotropine to infusible resins. Orig. art. has: no graphics.

ASSOCIATION: Kafedra khimicheskoy tekhnologii Moskovskogo gosudarstvennogo universitet (Department of Chemical Technology, Moscow State University)

SUBMITTED: 10Apr64

ENCL: 00

SUB CODE: MT, OC

NR REF SOV: 000

OTHER: 000

Card 2/2

PETROV, K.A., NIKANTSEV, E.No., GOLITSOVA, R.O., KORNEEV, A.V.

Phosphorus-containing polymers. Part 12: Synthesis of
polymeric analogs of phosphorus-containing insecticides.
Vysokom.sped. no. 5: 22-23 My '64. MIRA 17:6

L 10683-B5 EWT(M)/EPF(G)/EWP(J) Pc-4/Pr-4 RM

ACCESSION NR: AP4045417

S/0190/64/006/009/1545/1549

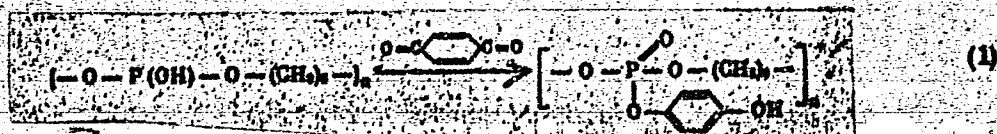
AUTHOR: Petrov, K. A.; Nifant'yev, E. Ye.; Gol'tsova, R. G.

TITLE: Synthesis of polyphosphite-based neutral polyphosphates and amidophosphates

SOURCE: Vysshemolekulyarnyye soyedineniya, v. 6, no. 9, 1964, 1545-1549

TOPIC TAGS: polyphosphate, neutral polyphosphate, amidophosphate, polyphosphite, phosphorylated polymer

ABSTRACT: Neutral polyalkylenephosphates (mol. wt. 12000 - 15000 determined from light dispersion in dimethylformamide) were prepared either by the chlorination of polyalkylenephosphites (mol. wt. 30000) to polyalkylenechlorophosphates with subsequent esterification of the products with alcohols, by the reaction of polyphosphites with p-quinone:

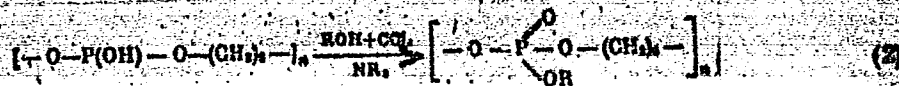


Card 1/3

L 10683-65

ACCESSION NR: AP4045417

or by the reaction of polyalkylenephosphites with alcohols in the presence of carbon tetrachloride and a tertiary amine:



By substituting primary or secondary amines for tertiary amines in (2), polyalkylene-amidophosphates were also prepared. The 12 products discussed are: polyhexamethylenechlorophosphate, poly-3-oxaamylenechlorophosphate, polyhexamethyleneethylphosphate, the p-hydroxy phenyl ester of polyhexamethylenephosphoric acid, polyhexamethylenebutylphosphate, neutral polyhexamethylenephosphate, the beta-cynceethyl ester of polyhexamethylenephosphoric acid, the beta-diethylaminoethyl ester of polyhexamethylenephosphoric acid, the dibutylamide of polyhexamethylenephosphoric acid, the hexamethylenediamine-based amide of polyhexamethylenephosphoric acid, the delta-aminoethylamide of polyhexamethylenephosphoric acid, and the diethylammonium salt of the p-carboxyphenylamide of polyhexamethylenephosphoric acid. The procedures and some characteristics of each individual product are presented. Reaction (2) was found to be a more suitable process. Orig. art. has: 3 chemical equations.

Card 2/3

L 10683-65

ACCESSION NR: AP4045417

ASSOCIATION: None

SUBMITTED: 23Feb63

ENCL: 00

SUB CODE: 00

NO REF SOV: 005

OTHER: 001

Card 5/8

PETROV, K.A.; NIFANT'YEV, E.Ye.; KHORKHOYANU, L.V.; SHCHERBA, I.G.

Phosphites and phosphinites of triols and their derivatives. Zhur.ob.
khim. 34 no.1:70-77 Ja '64. (MIRA 17:3)

PETROV, K.A.; NIFANT'YEV, E.Ya.; SHCHEGOLEV, A.A.; TUSEYEV, A.P.

1,2,3,4-Diisopropylidenegalactose 6-methyl phosphinite. Zhur.ob.khim.
34 no.2:690-693 F '64. (MIRA 17:3)